

**NEVADA MODIFIED NET ACID GENERATION TEST PROCEDURE****SUMMARY**

July 2020

The Nevada Division of Environmental Protection, Bureau of Mining Regulation and Reclamation (Division) has determined the 2020 Nevada Modified Net Acid Generation (NV-NAG) Test Procedure to be an acceptable alternative for demonstrating potential acid generation (PAG) from barite, gypsum, alunite, and other similar type minerals. Sulfate-bearing minerals have been observed to exhibit on repeated occasions false positive PAG results when tested under the commonly used and industry accepted Nevada Modified Sobek Procedure (NMSP). This phenomena has been verified in the literature.

When the presence of sulfate-bearing minerals is suspect and an accurate laboratory PAG determination is necessary, the Division will require samples to be characterized using the Nevada NV-NAG test procedure, mineralogical characterization, and NMSP testing of suspect samples by a Nevada-certified laboratory. In addition, the operator is required to provide sample mineralogy, Nevada NV-NAG, and NMSP results to the Division.

Beginning 1 August 2020\*, the laboratory must be approved by the State of Nevada Bureau of Safe Drinking Water – Laboratory Certification Branch for the Nevada NV-NAG method. In the interim, the laboratory is authorized to perform NV-NAG testing, but must be actively working towards method approval.

**Procedure Summary**

The NV-NAG test involves the addition of 250 milliliters (ml) of 15% hydrogen peroxide ( $\text{H}_2\text{O}_2$ ) to 2.5 grams (g) of pulverized (95% minus 150 mesh) sample. Allow sample to react until ‘boiling’ or effervescence ceases. This may require the sample to be left overnight, at the longest. The entire sample is heated until gently bubbling for approximately 1-2 hours to remove excess  $\text{H}_2\text{O}_2$  and encourage release of inherent neutralizing capacity. Once the sample has cooled to room temperature, measure the pH of the solution (NAG pH).

A NAG pH less than 4.5 standard units (SU) will require repeating of the NV-NAG test on the filtered solid residue of the sample, repeat this process until the NAG pH is greater than or equal to 4.5 SU. Once the NAG pH is greater than or equal to 4.5 SU, titrate the solution to 7.0 SU, and add all NAG acidities to determine total NAG acidity in kg  $\text{H}_2\text{SO}_4$ /t.

Solution assays of sequential NAG solutions may be carried out by filtering solutions from each stage using No. 40 Whatman or finer filter paper, topping up the filtered solutions to the original 250 ml with deionized water to account for loss by evaporation, and analysis of solutions for metals (and sulfur [S]) by ICP-OES/MS.

**Principles**

The NAG test involves the addition of hydrogen peroxide ( $\text{H}_2\text{O}_2$ ) to a prepared sample of mine rock or process residue to oxidize reactive sulfide minerals, followed by measurement of the pH of the reaction solution to provide a total acid-producing potential for samples in which oxidation of sulfide sulfur (S) is incomplete (typically when pyritic S is greater than 0.7 to 1% S). Then titration of any net acidity produced.

## Comments

A temperature rise is commonly observed in NAG testing of sulfidic samples as a result of catalytic decomposition of peroxide by metal ions released during sulfide oxidation.

The pH of the 15% hydrogen peroxide solution should be checked prior to use to ensure it is greater than or equal to 4.5. To adjust the pH use a NaOH solution made up by adding 1 g NaOH to 100 mL of deionized water. A target pH of 4.5 but not greater than 6.0 is desired. Record the pH of the H<sub>2</sub>O<sub>2</sub> reagent for each batch.

## Chemicals

1. Hydrogen Peroxide (H<sub>2</sub>O<sub>2</sub>) 15%
2. Hydrochloric acid (HCl) solution, 0.1 Normality (N), certified grade (Fisher So-A-54 or equivalent).
3. Sodium hydroxide pellets (Fisher S320-10 or equivalent)
4. 0.5 N Sodium hydroxide (NaOH): Dissolve 20.0 grams (g) of NaOH pellets in carbon dioxide (CO<sub>2</sub>) free water and dilute to 1.0 liter. Protect from CO<sub>2</sub> in the air with ascarite tube. Standardize solution by placing 50 milliliters (ml) of certified 0.1 N HCl in a beaker and titrating with the prepared 0.5 N NaOH until a pH of 7.00 SU is obtained.

Calculate the Normality (N<sub>2</sub>) of the NaOH using the following equation:

$N_2 = (N_1 V_1) / V_2$ , where

$V_1$  = Volume of HCl used, ml;

$N_1$  = Normality of HCl used;

$V_2$  = Volume of NaOH used, ml;

$N_2$  = Calculated Normality of NaOH.

5. 0.1 N Sodium hydroxide (NaOH): Dilute 400 ml of deionized water with 100 ml of 0.5 N NaOH
6. pH 1.68 buffer solution
7. pH 4.0 buffer solution
8. pH 7.0 buffer solution
9. Deionized water

## Materials

1. Burette;
2. Hot Plate;
3. pH meter equipped with combination electrode;
4. Balance, accurate to 0.01 gram;
5. No. 40 Whatman or finer filter paper;
6. 500 ml conical flask

## Procedure

1. Prepare the sample to 95% minus 150 mesh.
2. Check the pH of the H<sub>2</sub>O<sub>2</sub> to ensure the pH is between 4.5 and 6 SU. If the pH is less than 4.5 SU, add diluted NaOH until the pH is greater than 4.5 SU. This step must be performed to negate a false positive result of the sample being identified as acid generating.
3. In a 500 ml flask combine 2.5 g ± 0.1 g of pulverized sample, or filter paper/solid residue from Step 6, with 250 ml of 15% H<sub>2</sub>O<sub>2</sub> (equilibrated to room temp). Allow sample to react until 'boiling' or effervescence ceases. This may require the sample to be left overnight, at the longest.
4. Place sample on a hot plate at medium heat and gently boil for 1 to 2 hours or until the effervescing ceases. This allows removal of excess H<sub>2</sub>O<sub>2</sub> and encourages release of inherent neutralizing capacity. **The sample must not be allowed to boil dry;** add deionized water if necessary.
5. Allow the sample to cool to room temperature and record the final pH (NAG pH).
6. Filter the solid from the liquid using No. 40 Whatman or finer filter and retain.
7. Add deionized water to the liquid to bring it back to 250 ml.
8. If NAG pH is greater than 4.5, titrate the solution to pH 7.0 with 0.10 M of NaOH and record the volume. Report the titrated acidity as the NAG Acidity (NV-NAG).
9. If NAG pH is less than 4.5, titrate the solution to pH 4.5 with the appropriate molarity of NaOH (see table below) and record the volume.

NAG pH	NaOH Concentration
>2 SU	0.10 M
<2 SU	0.50 M

10. Then continue to titrate the solution to pH 7.0 with the same molarity of NaOH as before.
11. If NAG pH is less than 4.5, repeat Steps 2 through 9 until the NAG pH is greater than 4.5. Record volume of NaOH for each titration separately. Add all of the titrated NAG acidity to get a total NAG Acidity in kg H<sub>2</sub>SO<sub>4</sub> / ton sample.

## Calculations

1. Calculate NV-NAG using the following equation:

$$\text{NV-NAG (in kg H}_2\text{SO}_4\text{/ton)} = \frac{49(V)(N)}{W}$$

V = Volume of base NaOH titrated, in ml

N = Normality of the base NaOH, in moles per liter

W = Weight of the sample, in grams

Please note the calculation may need to be completed for each normality then be added together.

## Reporting Requirements

- 1 – Report NAG pH from Step 5, and each at sequential addition, as appropriate.
- 2 – Report NaOH volume (ml) used in Step 9 for each sequential addition.
- 3 – Report NaOH normality used in Step 9 for each sequential addition.
- 4 – Report total volume of NaOH titrated.
- 5 – Report NV-NAG in kg H<sub>2</sub>SO<sub>4</sub>/ton.

## References:

AMIRA International Ltd. (2002). ARD Test Handbook – Prediction and Kinetic Control of Acid Mine Drainage. Environmental Geochemistry International Pty. Ltd. and Ian Wark Institute, University of South Australia.

Warwick A. Stewart, Stuart D. Miller, and Roger Smart, *Advances in Acid Rock Drainage (ARD) Characterisation of Mine Wastes*, Paper presented at the 7th International Conference on Acid Rock Drainage (ICARD), March 26-30, 2006, St. Louis MO. R.I. Barnhisel (ed.) Published by the American Society of Mining and Reclamation (ASMR), 3134 Montavesta Road, Lexington, KY 40502.

Sobek et al, (1978), Field and Laboratory Methods Applicable to Overburden and Minesoils, EPA 600/2-78-054, 203pp.

\* The majority of the Nevada laboratories approved/certified under the “Mining Approval Program” have been approved for a similar version of this method since approximately 2017. The Nevada Modified Net Acid Generation Test Procedure standardizes sample grind size consistent with the NMSP and mandates the sequential addition as the minimum test protocol. The 1 August 2020 requirement corresponds to the laboratory certification renewal date and mandates the implementation date of modifications to existing certifications.